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Preparation Of PVP.BtB Thin Films by Spin Coating Technique and Study the Structural and Optical Properties for Optical Filters Application

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Abstract:

To fabricate optical filters, the pure Polyvinylpyrrolidone (PVP) polymer thin films doped with Bromothymol blue (BTB) dye at different doping concentrations (0.2, 0.4, and 0.6 %wt) were prepared by the spin-coating method. FE-scanning electron microscope examinations indicated that all the pure and doped thin films were highly homogeneous and almost free of morphological defects. X-ray diffraction examinations indicated that the pure PVP thin films initially had an amorphous structure, which transformed into a polycrystalline structure upon doping with BTB dye; the crystal size was 41.1 nm at 0.2%wt BTB and increased to 60.4 nm at 0.6 %wt BTB. Optical measurements indicated that the doped thin films have selective ability at wavelengths of 406 and 620 nm. The studies also indicated that the prepared films have indirect electronic transitions and that the optical energy gap decreases with increasing BTB doping concentration. From the above results, the prepared films can be effective and scientific in optical filter applications, as they can select wavelengths located at 406 and 620 nm of the visible spectrum.

Keywords: Bromothymol blue, optical filter, polyvinylpyrrolidon, spin coating.

Note: The research is based on an M.A thesis or a PhD dissertation (if any). No

Introduction:

Polyvinylpyrrolidone, also known as povidone or polyvidone, is a polymer, highly soluble in water, obtained from the monomer N-vinylpyrrolidone. In addition to its solubility in water, it is also soluble in many other polar solvents. It has the ability to absorb water from atmospheric humidity. It has wetting properties, and the ability to easily form thin films, making it suitable for use in paints and coatings. PVP is a branched polymer. Its density is 1.2 g/cm^3 and its melting point is 150-180°C [1,2]. PVP in its pure form is completely harmless; not only is it harmless, but it has been used as a blood plasma expander in trauma victims since the mid-20th century. Polyvinylpyrrolidone bonds with polar molecules very effectively due to its polarity. This property makes it highly effective in high-efficiency paper coatings and also in printer inks [3,4]. Bromothymol blue, triphenylmethane dye, by chemical structure belongs to the group of sulfophthaleins, $C_{27}H_{28}Br_2O_5S$, molar mass 624.38 g/mol. It is a finely crystalline powder from pink to violet color, slightly soluble in water and benzene, soluble in methyl and ethyl alcohols, diethyl ether and dilute alkali solutions. Obtained by the interaction of elemental bromine with thymol blue in glacial acetic acid [5,6]. Bromothymol blue is used in bacteriology for the preparation of nutrient media used in microbiological studies, visualization of cell walls or nuclei when examining them under a microscope, indication of respiratory activity or photosynthesis, and as a photosensitizer in copying equipment. The main area of application of bromothymol blue is as an acid-base indicator in titrimetric methods of chemical analysis. Depending on the acidity of the medium, the indicator changes its color from yellow in an acidic medium (up to pH 5.8) through grassy green in a neutral medium to blue in an alkaline medium (pH over 7.6). The color transition is associated with a change in the structure of the chromophore groups in the indicator molecule [7]. The growth in the production of water-soluble polymers in the chemical industry has created an opportunity to enhance and develop various technologies for disperse dyeing blended materials, thereby improving their performance properties [8]. Therefore, the introduction of new or improved technologies for dyeing polymers aims to reduce economic costs, enabling a broader application of the developed composition and enhancing competitiveness in producing functional binder [9]. Thin films of the required quality can be produced by several methods. Each method has advantages, disadvantages, challenges, and critical parameters to achieve uniform coating. Understanding the differences between deposition methods is necessary to properly select the appropriate method for the experimental needs. When selecting a method, it is important to consider scalability and compatibility with production. Fortunately, most methods are compatible with several solutions, so the operator usually has several options to choose from [10]. In spin coating, a solution is applied to a substrate, which is then spun at speeds up to 20,000 rpm. The centrifugal force generated by constant acceleration, combined with viscous drag and surface tension, causes the solution to be evenly distributed across the substrate. The film thickness is determined by the spinning speed. Spin coating produces homogeneous films, ranging from polymer blends to microcrystalline films, making it a highly efficient method. Spin coating has become the deposition standard in thin-film research and is often used in the processing of photoresists on sheets and in the production of thin-film electronic devices [11]. In this regard, this research presents the results of a study on PVP polymer thin films, pure and doped by BTB dye, and a study of the effect of this dye on the structural and optical properties of these films. The study also focuses on the preparation of an optical filter from this thin film due to the high selectivity of the BTB dye for some wavelengths.

Experimental Part:

A PVP polymer powder $(C_6H_9NO)_n$, with a molecular weight of 300,000 g·mol^{-1} and a purity of 98%, from (Jinan Future Chemical Industry Co.,Ltd) was used. High-purity BTB dye $C_{27}H_{28}Br_2O_5S$ from (Science Company) was used to dope the PVP polymer. Distilled water and ethanol were used as solvents to prepare the solutions of the above materials. To prepare pure and doped PVP thin films by the spin coating method, the PVP solutions with 3% wt were prepared by dissolving PVP in 30:70 ethanol: water. The thin films were then prepared using a spin coater at 2000 rpm at room temperature on glass substrates. The same steps were repeated after doping the polymer with different concentrations of BTB dye (0.2, 0.4, and 0.6 %wt). The structural properties of the prepared films were characterized using X-ray diffraction (XRD), type PhillipsXpert (Holland), while the morphological structure was determined using a FE-scanning electron microscope (FE-SEM), type (Tescan Mira3, France). The chemical composition and functional groups of the prepared samples were determined using an infrared spectrometer FTIR in the range of 500 to 4000 cm^{-1}. The optical properties represented by the absorbance and transmittance spectrum were measured using a Sp-3000 Plus UV-Vis spectrophotometer in the range of 300 to 800 nm.

Results and Discussion:

Given the importance of the effect of the surface morphology of thin films on the physical properties of these thin films, the morphology of the prepared PVP thin films and the effect of the BTB dye on them were studied using FE-Scanning electron microscope images. From Figure 1, it can be clearly seen that all the prepared PVP thin films have homogeneous surfaces and are almost free of defects and cracks, except for some rare clusters that appear scattered on the surfaces of these thin films. These images also clearly show that adding the BTB dye does not affect the morphology of the PVP thin films; even as its percentage in the PVP thin films increases, they remain highly homogeneous, indicating a high compatibility between the PVP polymer and the BTB dye.

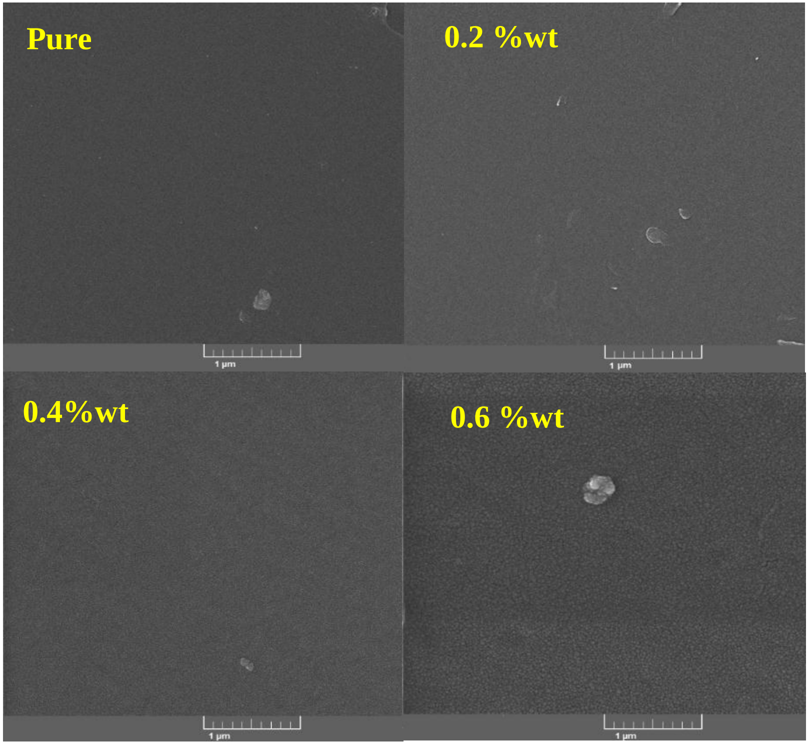

Figure 1. FE-SEM images of the PVP and PVP.BTB thin films.

X-ray diffraction was applied to compare the crystalline structure of the samples. The resulting XRD patterns are shown in Figure 2. The XRD pattern of pure PVP exhibits an amorphous nature, with broad peak around 2θ = 22°, clearly indicating the absence of a crystalline structure [12]. This phenomenon is common in polymeric materials, where short-range polymer chains dominate the crystalline system of the sample. This means that the above preparation conditions cannot be used to prepare polycrystalline or crystalline PVP thin films. It can be observed that the addition of BTB leads to the transformation of the PVP samples from amorphous to a polycrystalline structure; the higher the proportion of BTB, the more the crystallinity increases. Two peaks of growth were observed at 2θ = 11.7° and 22.4°, and the intensity of these peaks increased with a higher BTB weight percentage. These peaks are attributed to the crystalline phases of the BTB dye and the polymer-dye matrix, which indicates that the dye has been successfully incorporated into the polymer through its interaction with the polymer chains [13]. It was also observed that the crystal size of the prepared films increases from 41.1 nm at 0.2%wt BTB and increased to 60.4 nm at 0.6 %wt BTB. This indicates the enhancement and modification of the crystalline structures after doping, thus enabling the samples to be used in practical applications.

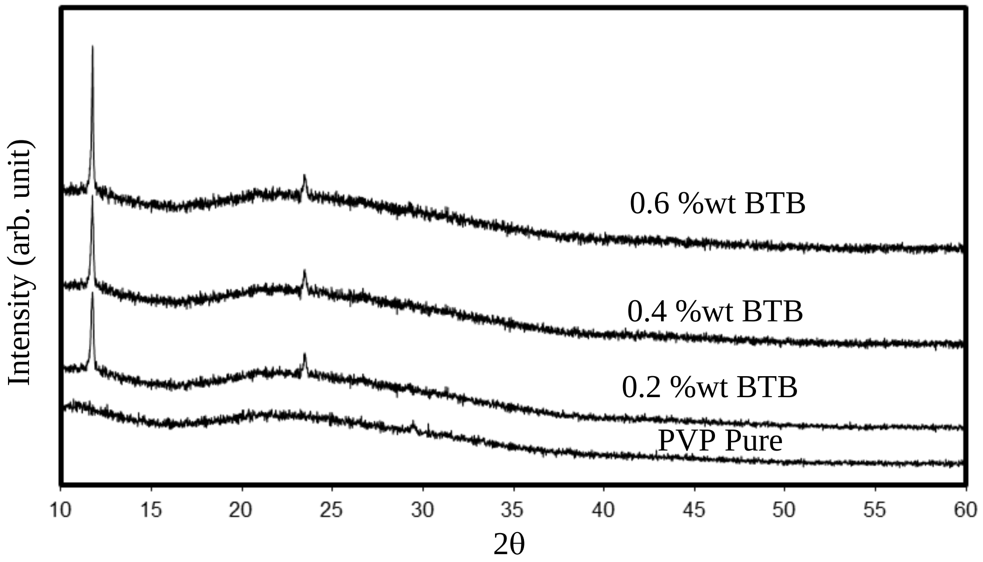

Figure 2. X-ray diffraction pattern of PVP and PVP.BTB thin films.

Pure PVP and PVP.BTB thin films were studied using FTIR infrared spectroscopy to confirm and identify the functional groups and bonds present in them, as well as the fact that partial interactions play an important role in the properties of the prepared samples. The spectrum of pure PVP thin films (Figure 3) showed a high consistency with the standard spectra of this polymer, with several bands appearing at 2969-2930 cm^{-1} indicating the C-H bond, 1656 cm^{-1} corresponding to the C=O bond, and 1286 cm^{-1} corresponding to the C-N bond [14]. When pure PVP is doped by BTB dye, and since both materials are capable of forming hydrogen bonds with water molecules, they can also be linked via C=O (carbonyl) or phenolic –OH groups, a noted an expansion in the C=O bond at wavenumber 1656 cm^{-1}, as well as an increase in the width of the O-H band at wavenumber 3465 cm^{-1} [15, 16].

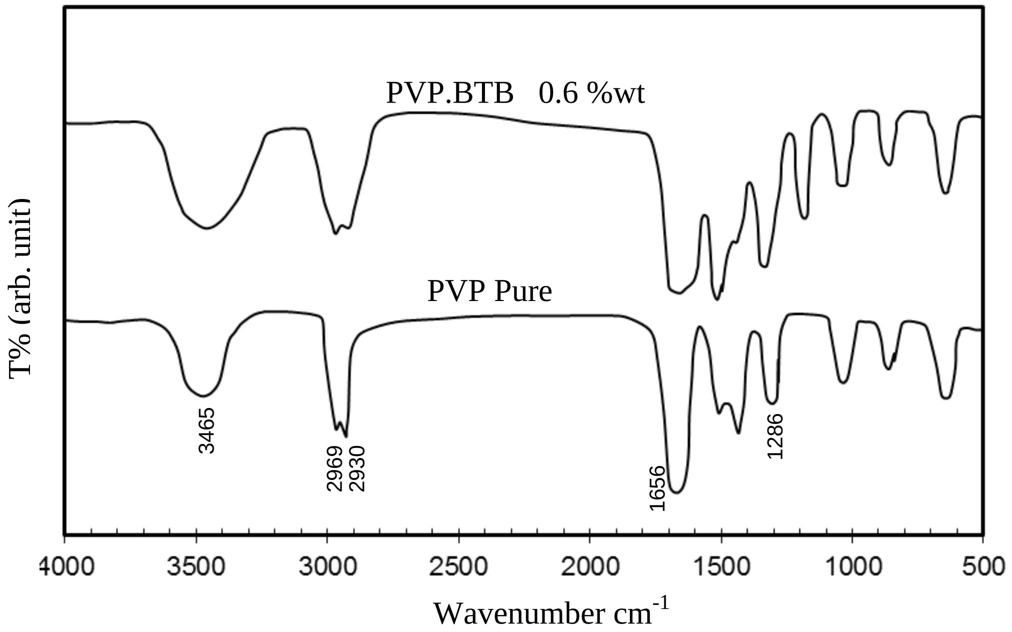

Figure 3. The FTIR spectra of Pure PVP and PVP.BTB at 0.6%wt.

Figures 4 and 5 show the transmittance and absorbance curves as a function of wavelength for pure PVP and PVP.BTB thin films. From Figure 4 for pure samples, a sudden increase in transmittance values is observed with increasing wavelength in the range 275-400 nm, then the curve begins to stabilize almost after the wavelength 400 nm, meaning that PVP films have very high transmittance for wavelengths greater than 400 nm, as the above result helps in choosing the appropriate applications for PVP thin films. For the PVP.BTB thin films, two peaks were observed: one at 400 nm, due to π-π^* transitions in the conjugated bonds in the aromatic ring of the BTB dye, and the other peak at 620 nm resulted from electronic transitions resulting from the delocalization levels in the energy band structure [17]. It was also observed that the intensity of these peaks increased with increasing BTB dye doping concentration, which is attributed to the increase in excited molecules [18]. As for the absorbance curves, they behave in a completely opposite manner to the transmittance curves, as the absorbance of thin PVP films increases significantly with the increase in the BTB dye concentration, as is clear in Figure 5.

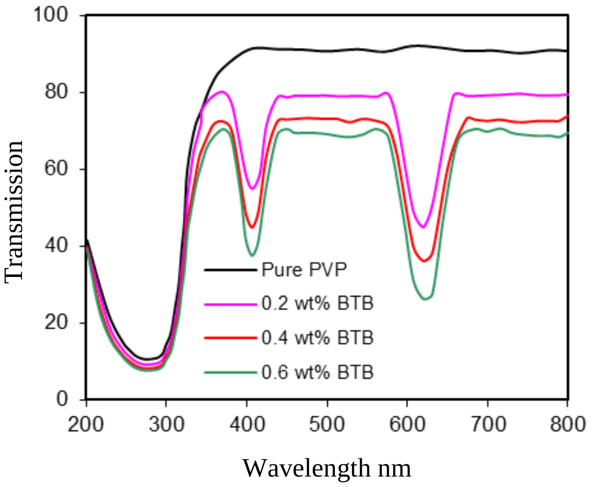

Figure 4. The variation of transmission spectra with wavelength of pure PVP and doped with different wt% of BTB dye thin films.

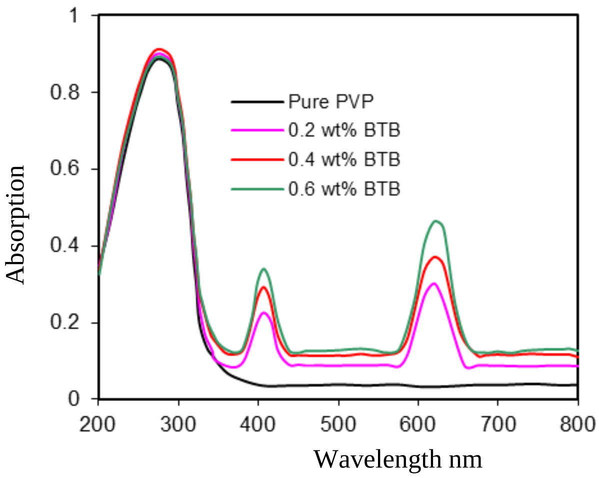

Figure 5. The variation of absorption spectra with wavelength of pure PVP and doped with different wt% of BTB dye thin films.

The absorption coefficient was calculated from the equation below for each wavelength, as well as the photon energy at each wavelength [19]:
$a = 2.303\ (A/t)$ ………………..(1)
A (absorption), t (thickness of thin films).

Figure 6 shows the change in the absorption coefficient with photon energy for pure PVP thin films doped with BTB dye. It is noted that its value starts at approximately 10^{3} cm^{-1} and increases with increasing photon energy to reach the highest value of approximately 5□10^{4} cm^{-1}. This helps to predict the occurrence of indirect electronic transitions within the range of the above energies, and this does not prevent the occurrence of direct electronic transitions [20]. It is noted in Figure 6 that the absorption coefficient clearly increases with increasing the concentration of BTB dye.

Figure 7 shows electronic transitions in PVP and PVP.BTB thin films, and it appears that the allowed indirect transitions are the most applicable in the prepared samples, based on the Tauc's equation [21].

$$\alpha h\nu = A\left(h\nu - E_g\right)^n \quad \text{…………………(2)}$$

A is a constant and n an exponential factor that determines the nature of the transitions (1/2 direct transitions and 2 for indirect electronic transitions). It is noted from the figure that the optical energy gap of the prepared films decreases with increasing doping concentration of BTB dye, from 3.7 eV for pure PVP thin films to 3.61 eV for thin films doped with 0.6 %wt BTB. This is due to the increase in the number of π-π* transitions present at the localized states inside the energy gap, which causes a decrease in the optical energy gap [22]. Based on the above results, it can be concluded that doping with BTB dye leads to improved absorption of thin films in a specific range of visible light, making them suitable for optical filter applications.

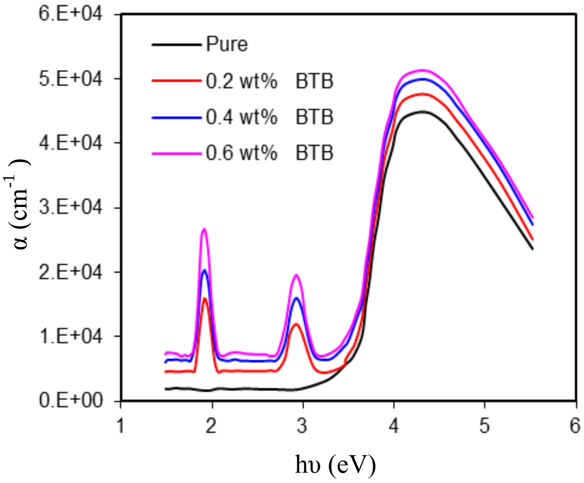

Figure 6. The variation of α with hυ of pure PVP and doped with different wt% of BTB dye thin

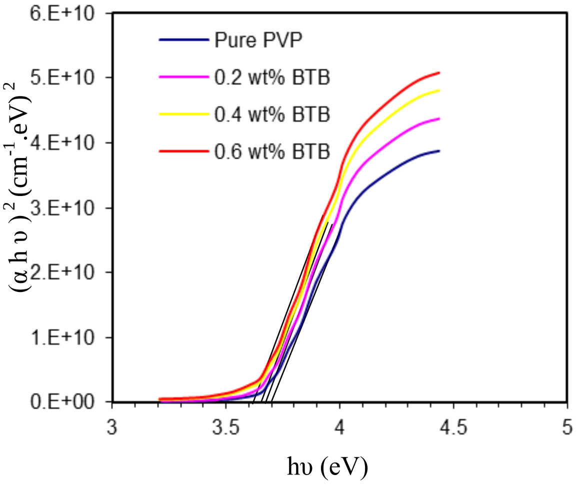

Figure 7. The variation of $(\alpha h\upsilon)^2$ with hυ of pure PVP and doped with different wt% of BTB dye thin films.

Extinction coefficient (k) values depend on the density of free electrons in the material and the presence of structural defects. The extinction coefficient for all prepared films was calculated using the relationship below [23] [21] :-

$$k = \frac{\alpha\lambda}{4\pi} \quad \ldots\ldots\ldots\ldots\ldots\ldots(3)$$

Figure 8 shows the change in extinction coefficient as a function of wavelength for PVP and PVP.BTB thin films. It is noted from the figure that pure samples have a high extinction coefficient at short wavelengths and decrease, then stabilize, at long wavelengths. However, samples doped with BTB dye show irregular behavior with increasing wavelength, where the extinction coefficient clearly increases at some wavelengths due to the localized levels generated within the energy gap when BTB dye is added. On the other hand, an increase in the extinction coefficient is noted with increasing doping concentration. Therefore, it can be concluded that the doping process improves electromagnetic radiation absorption and thus makes it suitable for applications requiring high visible light absorption.

The refractive index n is defined as the ratio of the speed of light in a vacuum to its speed within the material. The refractive index n depends on several factors, including the type of material and its crystal structure. The refractive index also changes depending on changes in grain size, even if the material has the same crystal structure. The refractive index was calculated using the relationship below [23] [21]:

$$n=\left[\frac{4R}{(R-1)^{2}}-k^{2}\right]^{1/2}-\frac{(R+1)}{(R-1)} \qquad \ldots\ldots\ldots\ldots\ldots\ldots(4)$$

R (reflectivity).

Figure 9 shows the change in the refractive index values as a function of the wavelength of PVP and PVP.BTB thin films. It is noted in the figure that the refractive index of the pure films is relatively high at ultraviolet wavelengths and begins to decrease with increasing wavelengths in the visible range until it is almost stable. This pattern is a common behavior in polymers and is consistent with the normal dispersion law. For thin films doped with BTB dye, a general increase in the refractive index value was observed with increasing doping concentration, as well as the appearance of peaks associated with the dye's absorption at corresponding wavelengths. This indicates that the addition of BTB dye affects not only the absorbance but also the light scattering properties of the prepared thin films.

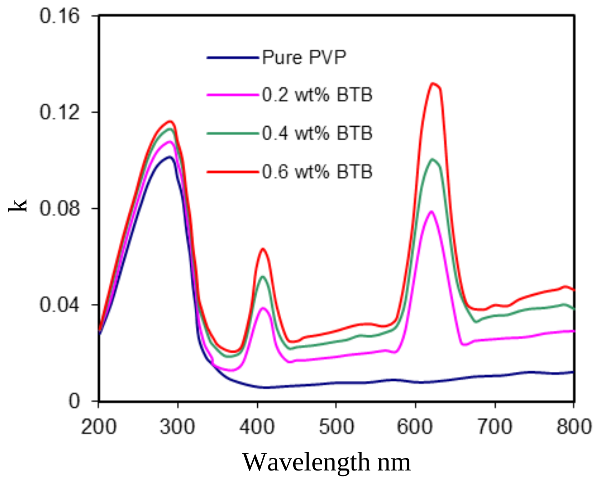

Figure 8. The variation of k with wavelength of pure PVP and doped with different wt% of BTB dye thin films.

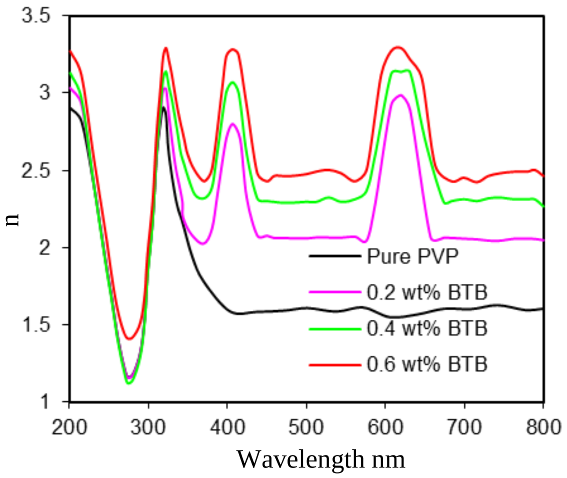

Figure 9. The variation of n with wavelength of pure PVP and doped with different wt% of BTB dye thin films.

From the above results, the prepared films can be effective and scientific in optical filter applications, as they can select specific wavelengths (402 & 620 nm) from the visible spectrum.

Conclusion:

It is concluded from the present work that thin films of pure PVP polymer doped with BTB dye can be successfully prepared using the spin coating method. These thin films have high morphological homogeneity, depending on FE-SEM results, as the addition of the BTB dye does not affect the morphology of the prepared films. It was also shown that the addition of the BTB dye to the PVP polymer leads to a transformation of the crystal structure from an amorphous to a polycrystalline state, which is an important point in changing the properties of the prepared PVP.BTB thin films, depending on X-ray diffraction results. The optical measurements clearly indicated that an optical filter for specific wavelengths 402 and 620 nm was obtained by doping the PVP polymer with the BTB dye, highlighting the potential use of this filter in optical devices.

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تحضير أغشية PVP.BTB الرقيقة بتقنية الطلاء الدوراني ودراسة الخصائص الهيكلية والبصرية لتطبيقات المرشحات البصرية

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مستخلص البحث:

لتصنيع المرشحات الضوئية، تم تحضير أغشية رقيقة من بوليمر بولي فينيل بيروليدون (PVP) النقي والمشوب بصبغة البروموثيمول الزرقاء (BTB) عند تركيزات مختلفة (0.2, 0.4, و 0.6٪ wt) بطريقة الطلاء الدوراني. أشارت فحوصات تاثير المجال-المجهر الإلكتروني الماسح إلى أن جميع الأغشية الرقيقة النقية والمشوبة كانت متجانسة للغاية وخالية تقريبًا من العيوب المورفولوجية. بينما أشارت فحوصات حيود الأشعة السينية إلى أن أغشية PVP الرقيقة النقية كان لها بنية غير متبلورة، تحولت إلى بنية متعددة البلورات عند التشويب بصبغة BTB، كان الحجم البلورة للاغشيو المحضرة 41.1 نانومتر عند 0.2٪ wt من BTB وازداد إلى 60.4 نانومتر عند 0.6٪ wt من BTB، أشارت القياسات الضوئية إلى أن الأغشية الرقيقة المشوبة لها قدرة انتقائية عند أطوال موجية 406 و620 نانومتر. أشارت القياسات أيضًا إلى أن الأغشية المحضرة لها انتقالات إلكترونية غير مباشرة وأن فجوة الطاقة الضوئية تتناقص مع زيادة تركيز التشويب بصبغة BTB. ومن خلال النتائج المذكورة أعلاه، يمكن للاغشية الرقيقة المحضرة أن تكون فعالة وعلمية في تطبيقات المرشحات البصري للأطوال الموجية الواقعة عند 406 و620 نانومتر من الطيف المرئي.

الكلمات المفتاحية : صبغة البروموثيمول الزرقاء، مرشح بصري، بولي فينيل بيروليدون، طلاء الدوران.

ملاحظة : هل البحث مستل من رسالة ماجستير او اطروحة دكتوراه ؟ كلا